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PATENT SPECIFICATION

NO DRAWINGS

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COMPLETE SPECIFICATION

Improvements in or Relating to Mould Release Agents

We, SOCIETE INDUSTRIELLE DES SILICONES, a body corporate organised under the laws of the French Republic, of 10 Av. Franklin D. Roosevelt, Paris 8e, France, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to mould release agents, and to a method of treating moulds, and it relates more particularly to new hardenable compositions having an organopolysiloxane resin base, intended for the internal coating of moulds with a view to facilitating the removal of articles from the moulds.

To facilitate the removal from the mould of articles, especially those made by pressure moulding, more particularly articles made of rubber or other plastic materials, it is known to use, as mould release agents, soaps of the type of stearates or other soaps, special oils, and liquid organo-siloxane compounds or emulsions or suspensions of the said liquid compounds. It is also known to use resinsifiable organo-siloxane compounds. However, the film obtained on the moulds by means of these mould release agents is quickly carried away by the moulded articles and must be renewed very frequently.

It is an object of the invention to provide improved mould release agents which adhere well to the mould and which make it possible to overcome this disadvantage.

According to the invention a mould release agent comprises a hardenable mixture of an organopolysiloxane resin and a titanium compound having the formula:—

$Ti_mO_{m-1}(OR^1)_{2(m+1)-p}(RCOO)_p$,
 m and p being integers and p being less than than $2(m+1)$ in which OR^1 is an alkoxy radical and $RCOO$ is an acyloxy radical and wherein the ratio of Ti/Si is from 0.001 to 1.

The organic radicals of the organopolysiloxane may include methyl, ethyl or other alkyl or phenyl or other aryl radicals. The organopolysiloxane may also contain silicon-bonded alkoxy radicals such as methoxy or ethoxy, or acyloxy radicals, such as acetoxyl. The resin can also contain silicon-bonded hydroxy radicals. The ratio of the non-hydrolysable organic radicals to the silicon is preferably between 1 and 2. 45

If for example m is equal to 3 and p is less than $m+1$, the product is a liquid which is difficult to obtain in the pure state. When $p=m+1$, the product is crystalline and easily obtained in the pure state. When p is between $m+1$ and $2(m+1)$, the product is again solid but it is of indefinite composition and is difficult to purify. 50

The invention extends to a method of treating a mould to facilitate removal of moulded articles therefrom, which comprises applying to the moulding surface of the mould a coating of the mould release agent according to the invention and allowing the coating to harden. 55

The mould release agents according to the invention can be applied to the moulds by any convenient methods for example with a brush by immersion or preferably by spraying with a spray gun or by means of an aerosol container, after dilution in "Freon" (registered Trade Mark). 60

In the latter case, ordinary aerosol containers can be used since the titanium compounds described are comparatively stable with respect to hydrolysis, and there is therefore no danger of the valve of the container becoming stopped up under the action of traces of moisture. 70

After being applied to the mould the mould release agent according to the invention can be hardened with the aid of heat. For this purpose the mould release agent can be applied to the mould in the cold, after which 80

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the mould can be heated to cause hardening of the mould release agent. It is however generally more convenient to apply the mixture according to the invention to the pre-heated mould, so that the mould is at an elevated temperature when the coating of mould release agent is applied thereto. The hardening time depends on the temperature, being less at higher temperatures. For example, complete hardening of the mixture generally requires about 30 to 45 minutes at 120° C, and only 5 minutes at 150° C, while at ambient temperatures hardening requires about 24 hours. It is generally preferred to carry out the hardening at temperatures similar to those used for the actual production of the moulded articles, more especially in the case of the moulding of natural or synthetic rubber or thermosetting resins.

The mixture according to the invention readily wets the surface of the mould and gives a very uniform and continuous film which solidifies quickly to give a hard, very smooth and resistant coating which adheres strongly to the surface of the mould. Articles of rubber or of plastic materials moulded in moulds which have been coated with the mould release agent according to the invention can be readily removed from the moulds because the coating does not adhere to the moulded materials. Moreover, the mould release agent according to the invention is so resistant and adheres so well to the mould that a considerable number of mouldings can be carried out without renewing the coating.

The following examples illustrate the production and use of the mould release agent according to the invention:—

EXAMPLE 1
The following substances are mixed in the cold:—

70 g of an organopolysiloxane resin containing silicon-bonded methyl radicals in the ratio to silicone of 1.25 and ethoxy radicals in the ratio to silicone of 1;
30 g of white spirit; and
12.2 g of the compound $Ti(OC_2H_5)_3(CH_3COO)$ containing 11.45% of titanium.

The liquid obtained is sprayed on a mould made of steel or of aluminium alloy heated to 150° C. It wets the surface of the mould well and gives a very uniform and continuous film. After 5 minutes at this temperature, a hard, very smooth brilliant coating is obtained, and a very large number of articles made of a composition having a rubber base, such as the composition used for shoe soles, can be moulded, removal of the articles from the mould remaining easy throughout the whole series of mouldings.

EXAMPLE 2

The same mixture of ingredients as in Example 1, in the amounts stated, is diluted with 670 g of "Freon". An aerosol container is filled with the solution and spraying is carried out on a steel or aluminium alloy mould heated to 150° C, as in Example 1. A brilliant, very hard coating is obtained having remarkable anti-adhesion properties in relation to moulded articles.

EXAMPLE 3

The following substances are mixed in the cold:—

70 g of an organopolysiloxane resin containing silicon-bonded methyl radicals in the ratio to silicone of 1.25 and ethoxy radicals in the ratio to a silicone of 1;

0.001 to 1.
30 g of white spirit; and
9.9 g of the compound $Ti_2O_3(OC_2H_5)_4(CH_3COO)_2$.

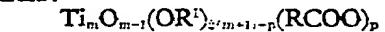
The liquid obtained is applied to a mould of steel or of aluminium alloy heated to 150° C. The liquid wets the surface of the mould well and gives a uniform and continuous film. After 5 minutes at this temperature, a hard brilliant smooth coating is obtained, and with the mould thus coated it is possible to mould a very large number of plates or other articles, such as soles or plugs, having a base of rubber compositions, from the more or less loaded mixtures entering into the composition of such articles. The moulded articles can be very easily removed from the mould, even after a prolonged series of moulding operations.

EXAMPLE 4

The same composition as that used in Example 3 is diluted with 670 g of "Freon". An aerosol container is filled with this solution and spraying is carried out on a mould made of steel or of aluminium alloy heated to 150°, as in Example 3. There is similarly obtained a brilliant, very hard smooth coating having remarkable anti-adhesion properties.

WHAT WE CLAIM IS:—

1. A mould release agent comprising a hardenable mixture of an organopolysiloxane resin and a titanium compound having the formula:—



m and p being integers and p being less than $2(m+1)$ in which OR^1 is an alkoxy radical and $RCOO$ is an acyloxy radical and wherein the ratio of Ti/Si is from 0.001 to 1.

2. A mould release agent according to claim 1 comprising a solvent or diluent.

3. A mould release agent according to claim 2 wherein the solvent or diluent is white spirit.

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4. A mould release agent according to any preceding claim wherein the alkoxy radical is a methoxy, ethoxy or butoxy radical.
5. A mould release agent according to any preceding claim wherein the acyloxy radical is an acetoxy radical.
6. A mould release agent according to any preceding claim wherein the organic radicals of the organopolysiloxane resin include methyl, ethyl or other alkyl, or phenyl or other aryl radicals.
7. A mould release agent according to any preceding claim wherein the ratio of the non-hydrolysable organic radicals to silicon in the organopolysiloxane resin is from 1 to 2.
8. A mould release agent according to any preceding claim wherein the organopolysiloxane also contains silicon-bonded methoxy, ethoxy or other alkoxy radicals.
9. A mould release agent according to any one of the preceding claims wherein the organopolysiloxane resin also contains silicon-bonded acetoxy or other acyloxy radicals.
10. A mould release agent according to any preceding claim wherein the organopolysiloxane resin also contains silicon bonded hydroxy radicals.
11. A mould release agent according to any preceding claim wherein m is greater than unity and p is equal to $(m+1)$.
12. A method of treating a mould to facilitate removal of moulded articles therefrom, which comprises applying to the moulding surface of the mould a coating of a mould release agent according to any one of the preceding claims and allowing the coating to harden.
13. A method according to claim 12 wherein the mould release agent is applied by dissolving it in a solvent and spraying the solution on the surface to be treated.
14. A method according to claim 13, wherein the solution is sprayed from an aerosol container.
15. A method according to any one of claims 12 to 14 wherein the coating is hardened with the aid of heat.
16. A method according to claim 15 wherein the mould is at an elevated temperature when the mould release agent is applied thereto.
17. A method according to claim 15, wherein the mould is heated after application of the coating of mould release agent.
18. A method according to any one of claims 14 to 16 wherein hardening of the coating is carried out at temperatures similar to those used for the moulding of the articles.
19. A mould release agent substantially as herein described.
20. A method of treating a mould to facilitate removal of moulded articles therefrom, substantially as herein described.

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